EXPERIMENTAL INVESTIGATION OF ULTRAHIGH VACUUM ADHESION AS RELATED TO THE LUNAR SURFACE

NINETEENTH QUARTERLY PROGRESS REPORT

1 JULY 1969 THROUGH 30 SEPTEMBER 1969

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Prepared for:
NASA Office of ADvanced Research
and Technology
Washington, D.C.

Contract NAS7-307
Date of Issue:
26 June 1964

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WESTERN DIVISION
SANTA MONICA, CALIFORNIA 90406

ABSTRACT

New, slower acting etchants, applied to cleaved orthoclase crystals, have brought out in greater detail the dislocation structures along the interface of perthite inclusions. A differentiation between microcracks in orthoclase and deeply etched dislocation etch pit arrays across microperthites is postulated. Preferential etching of naturally deformed olivine revealed microscopic polygonization of dislocations producing small angle tilt boundaries. Improved non-destructive replication for electron microscopy resulted in excellent reproduction of crystal surface structures and etch figures. A modified knife and anvil has been developed and tested for cleaving CaF_2.

TABLE OF CONTENTS

1.0 Introduction

1.1 General

This report presents a summary of work accomplished during the period 1 July 1969 through 30 September 1969, on the study of the ultrahigh vacuum adhesion of silicates as related to the lunar surface. This work is being conducted for the Office of Advanced Research and Technology, National Aeronautics and Space Administration, under Contract NAS7-307.

1.2 Objectives

The general purposes of this program are (a) to obtain quantitative experimental data concerning the origin of the electrical charge distribution produced by the ultrahigh vacuum fracture of various silicate minerals and model materials, (b) to achieve an understanding of the mechanisms responsible for the observed charge stability in relationship to the lunar environment, (c) to apply etching techniques to a variety of possible lunar silicates including shock-loaded and/or radiation damaged materials, (d) apply the results to further understanding the possible effects of environment on their structure in preparation for analyzing the lunar materials returned by Apollo, and (e) attempt to obtain information concerning bonding of crystal grains in polycrystalline rocks.

1.3 Approach

The approach used during this report period has been (1) to cleave silicates at ultrahigh vacuum and measure the electrostatic charge distribution produced, (2) to obtain microchemical and microphysical profiles on the cleaved surfaces, and (3) to apply defect etching techniques to a variety of possible lunar silicates.

Approach (1) serves to provide information on the origin and dynamics of the observed electrostatic charging and stability in dielectrics cleaved in ultrahigh vacuum. Approach (2) serves to provide direct evidence of the relative roles played by composition gradients, dislocations, and stress patterns in electrostatic charging and the potential influence on stability of the lunar environment. Approach (3) serves to provide information about the structure of dielectrics including those which are shock-loaded and/or radiation damaged to aid in recognizing shock and radiation effects in the lunar sample returned by Apollo. Approaches (2) and (3) also serve to provide information on intergranular bonding in polycrystalline rocks which will provide information concerning the basic adhesion and cohesion mechanisms and how these compare with previous observations.

1.4 Items of Interest

An invited paper "Adhesional Behavior of Dielectric Surfaces" was presented at the Conference on Physics of Adhesion," University of Karlsruhe, Germany, July 1969. Another paper "Comments on Lunar Surface Adhesion" is to be published in the Proceedings of the Working Group on Extraterrestrial Resources.

In related work sponsored by NASA Houston, NAS9-8082, visits were made to the University of Chicago, Geophysical Sciences Laboratories, to discuss olivine and orthoclase defect etching with Dr. P. B. Moore, and to the General Electric Research Laboratory, Schenectady, New York to discuss fission track measurements with Dr. R. L. Fleischer. In a telephone discussion with Dr. C. B. Raleigh, who is with the United States Geological Survey, Menlo Park, California, he offered to provide mechanically deformed olivine for etching dislocations produced artifically under controlled conditions.

2.0 Introduction

2.1 Defect Etching

The naturally deformed olivine crystal, OL27A, described in the Seventeenth Quarterly Report (see Figure 7) was analyzed further. Optical photomicrography of aluminized etched surfaces and positive replication for electron micrography were utilized to study in detail the (010) face of the successively polished and etched crystal. Figure 1 shows polygonized boundaries between three olivine grains. Figure 2 illustrates a small angle tilt subboundary on the (010) olivine in the [001] direction. Large dislocation etch-pits with pointed bottoms are aligned parallel to the (001) plane.

The chemical etching survey of crystalline defects in silicate minerals continued utilizing preferential etchant combinations composed of hydrofluoric, hydrochloric, nitric, and citric acid combinations. A new series of etchants, previously reported in the Seventeenth Quarterly Report NAS7-307, containing hydrofluosilicic or hydrofluoroboric acids, were studied further. Pink orthoclase, cleaved along the (001) face, etched with a fluoroboric acid mixture for one minute, revealed dislocation etch pits along the perthite-orthoclase matrix. Deep microcracks, which normally appeared after five to ten seconds etch with the rapid HF etchants, appeared as a series of etch pits across the perthites, continuing in a straight line into the orthoclase matrix. Figure 3 and Figure 4 illustrate the etched (001) surface. The other interesting feature is the long narrow perthites (or another phase) with dislocation etch pits symmetrically opposed at the matrix interface, sometimes disconnected, thus not giving the appearance of a microcrack. Orthoclase, etched with a hydrofluoroboric acid etchant, containing a metal salt additive, shown in Figure 5, appeared to etch microcracks preferentially in the orthoclase matrix. This should be compared with deep etching of dislocations in the perthite-orthoclase matrix, along microcracks and joining symmetrical pits in narrow phases shown in Figure 6. This positive replica illustrates the etching action of previously reported etchant No. 6. (See Seventeenth and Eighteenth Quarterly Reports.)

A (0$\bar{1}$0) cleavage of orthoclase, etched thirty minutes in hydrofluosilicic acid is shown in Figure 7. The electron micrograph was made from a positive replica. Crystallographic directions and dislocation etch pits previously revealed witb faster etchants, appear in greater detail.

Natural radiation fission damage found in biotite mica, utilizing the No. 5 etchant, was reported previously. Figure 8 illustrates examples of fission tracks found after twenty hours of etching.

2.2 Electrostatic Charging

2.2.1 Run No. E-11 CaF_2 (111)

Pure CaF_2 is the first synthetic, model dielectric material which has been cleaved in our vacuum system. It was first reported in the previous quarterly report. It is cubic (Z = 4) space group Fm3m. Perfect {111} cleavage has been a problem because strain induced by the rotary motion UHV feedthrough check (compounded by the cleavage itself) led to spontaneous fracture inside the collar, some hours after cleavage.

The notch cleavage system used for brittle fracture of mineral silicates has not been very successful with CaF_2. The tendency to fracture along intersecting {111} faces produces uncontrolled steps, e. g., in E-11 steps 500 μ high. This high point exhibited a strong positive peak positioned approximately 120° from the cleavage slot (see Figure 2, Eighteenth Quarterly Report). A secondary maximum was found 120° from both of these points.

A modified anvil and chisel system was developed and tested in air. The anvil had a rounded saddle-shape to prevent sharp edges touching the crystal characteristic of the V-slotted anvil used with the silicates. A single edge razor blade was substituted for the chisel. Without using a notch, it was possible to get a perfect cleavage if the direction was $[\overline{11}2]$ in air. This system is now being used for UHV cleavage.

3.0 Discussion

3.1 Defect Etching

The slow etchants developed for silicates consisting of complex fluorine acids with additions of other inorganic acids and inhibiting agents, have produced interesting results when applied to feldspar single crystals. They appear to be more selective to dislocations, and the slower reaction rates makes etching easier to control than with a wide range of HF etchant combinations.[1] One of the new fluoroboric acid etchants seemed selective to the potassium phase; this remains to be confirmed. Some defects previously classified as microcracks, may be only polygonized dislocation arrays, relieving stresses between the albite perthites and the orthoclase matrix. Some deeper cracks, revealed by optical and electron microscopy on cleaved surfaces before etching, may have formed due to cleavage and fracture stresses.

Chemically etched (010) dislocations in mechanically deformed olivine were described by C. Young (Reference 1). Dislocation etching of naturally deformed olivine crystal OL27A (Fosterite 85%, Fayalite 15%) using a new etchant resulted in revealing dislocations on the (001) and (100) faces as well as the (010) face. The predominant feature brought out by etching is polygonization producing low angle grain boundaries and tilt sub-boundaries on the (010) plane in the [001] direction, Figure 1.

Smaller (2 sec. arc) tilt sub-boundaries in the [100] direction on the (010) plane were also observed. Large dislocation etch pits on the (010) plane showed the crystal symmetry, but varied in size and shape. Most were rectangular-like with pointed bottoms, the longer, curved sides of the rectangle being parallel to the (001) plane.

Grain boundary angle measurement, Figures 1 and 2 for example, is based on the existence of a single dislocation for each missing lattice plane (on one side of an edge dislocation). When two grains form a boundary with angle Θ between them, missing planes start at regular intervals indicated by the dislocation etch pits. If the lattice spacing along the grain boundary is b and the lattice spacing perpendicular to the boundary is d, then the dislocation interval is given by $\tan \Theta = d/nb$. The denominator is the observed average separation of the dislocations.

The chemical etchants developed here for revealing dislocations, deformation and radiation damage in silicate crystal structures will have important application in the microchemical and microphysical examination of lunar rocks and minerals.

4.0 Future Work

Deformation of shock-loaded and radiation-damaged crystal silicates obtained from Sedan Nuclear Crater, Nevada test site, will be examined by the chemical etching. Slow etchants developed for the feldspars, pyroxeues and olivine will be used in the study. Mechanically deformed olivine from United States Geological Survey studies (see Reference 2) will be chemically etched for comparison with naturally deformed olivine structures. More emphasis will be placed on calcic plagioclases in the etching study since they form a major fraction of lunar feldspars. Another important lunar mineral, ilmenite, will be studied also.

Work will be completed on model material cleavage. An attempt will be made to test the hypothesis that dislocations are introduced by the cleavage process and the observed electrical charge is associated with the resultant strain.

Reference 1. Chapman Young III. Dislocations in the Deformation of Olivine, American Journal of Science, Vol. 267, 1969.

Reference 2. C. B. Raleigh. Mechanisms of Plastic Deformation of Olivine. J. Geophys. Res. 73, 5391-5406 (1968).

ILLUSTRATIONS

Electron Micrographs

Figure 1 Positive replica of polygonized olivine boundaries on polished and etched (010) surface. Insert is an optical photomicrograph. Angles between adjacent grains are indicated.

Figure 2 Positive replica of small angle grain boundary on olivine (010) in the [001] direction. Note the aggregate of edge dislocations almost parallel to the gra-n boundary.

Figure 3 Positive replica of pink orthoclase, cleaved (010), magnification approximately 8,000X.

Figure 4 Positive replica of pink orthoclase, cleaved (010), magnification approximately 29,000X.

Figure 5 Positive replica of pink orthoclase, Section 9, cleaved (001), magnification approximately 8,000X.

Figure 6 Positive replica of white orthoclase, cleaved (001) magnification approximately 29,000X.

Figure 7 Positive replica of white orthoclase, cleaved (001), magnification approximately 29,000X.

Figure 8 Fission tracks in biotite mica. Optical photomicrographs with both reflected and transmitted light. Magnifications approximately 325X and 570X.

FIGURE 1

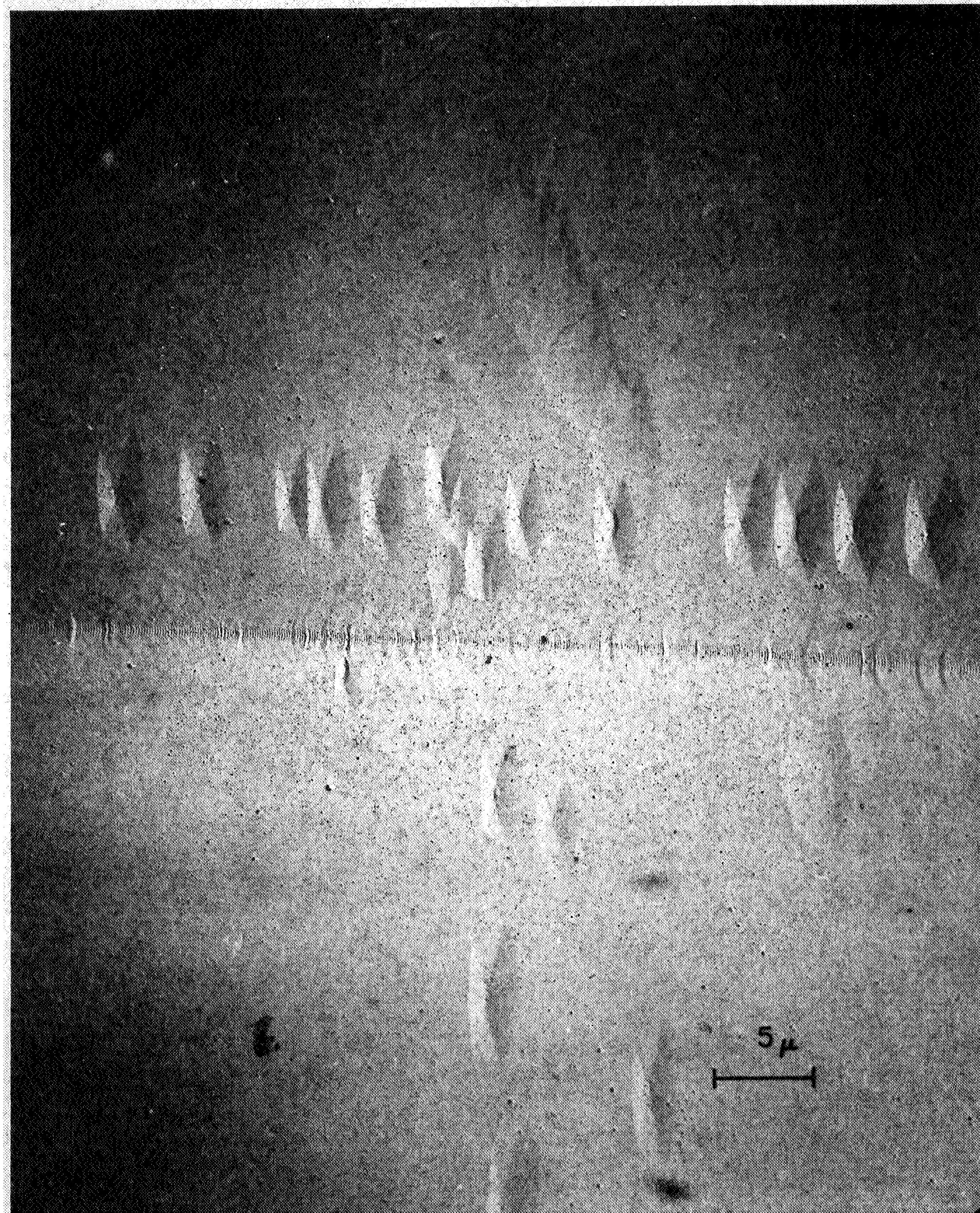

FIGURE 2

FIGURE 3

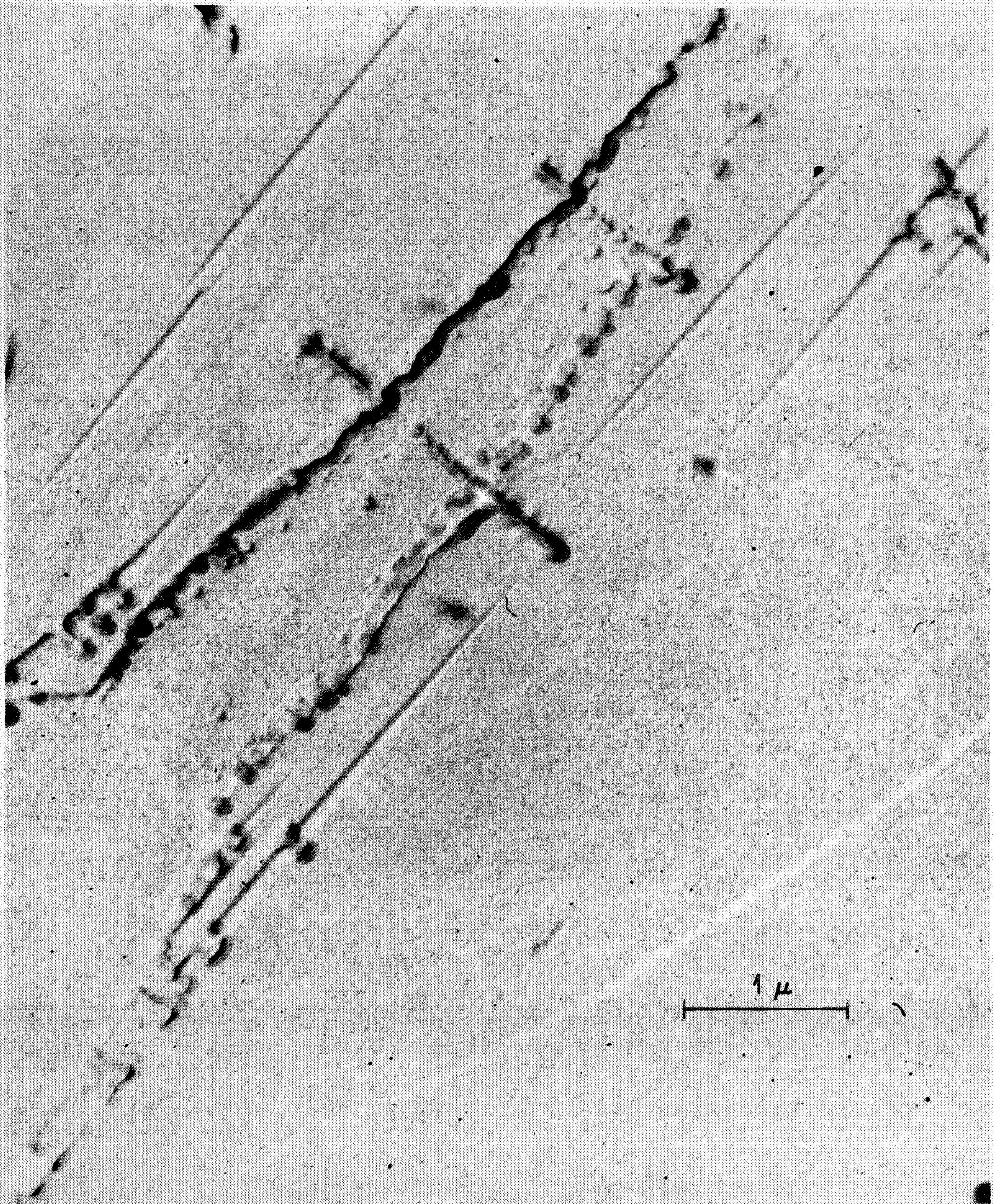

FIGURE 4

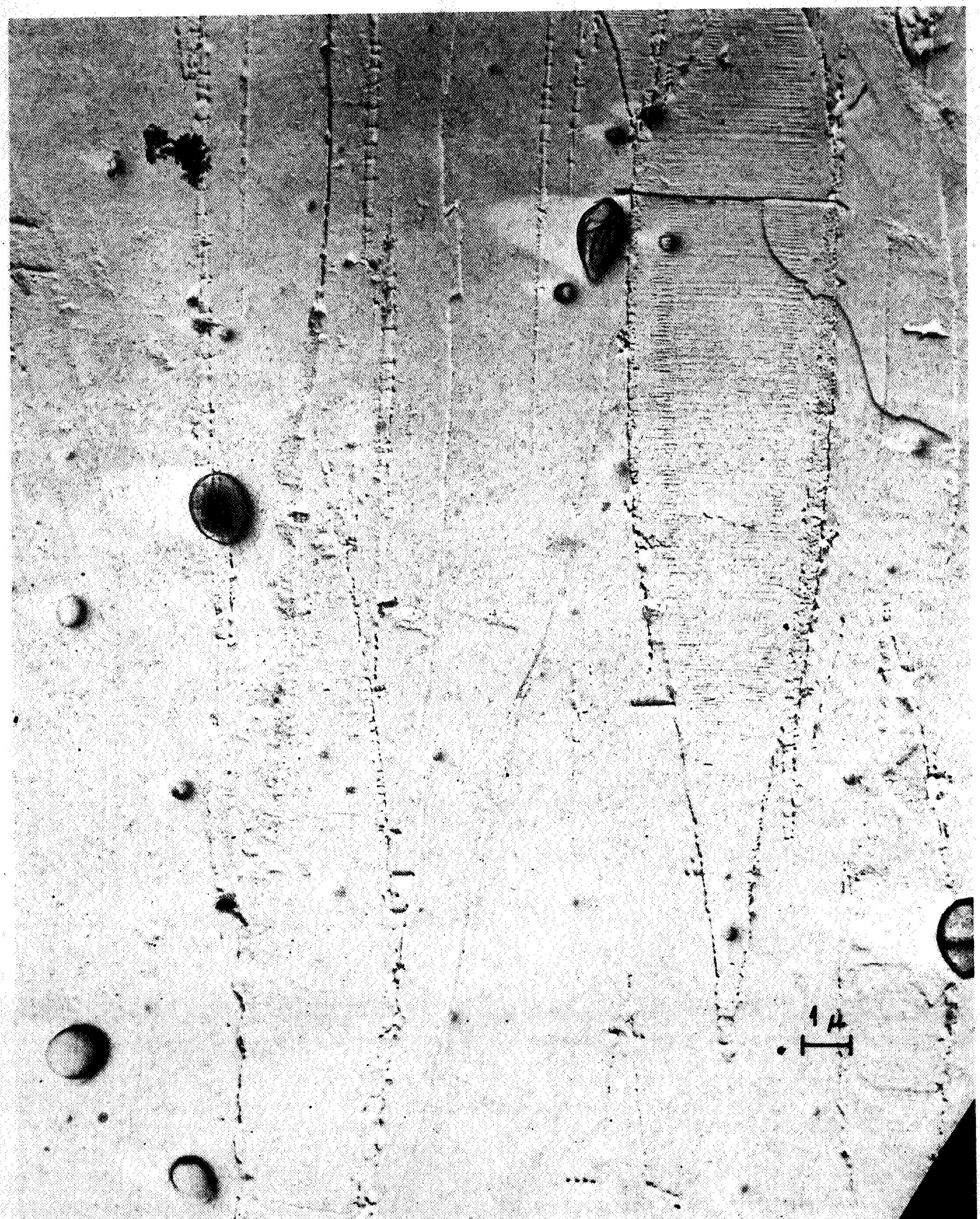

FIGURE 5

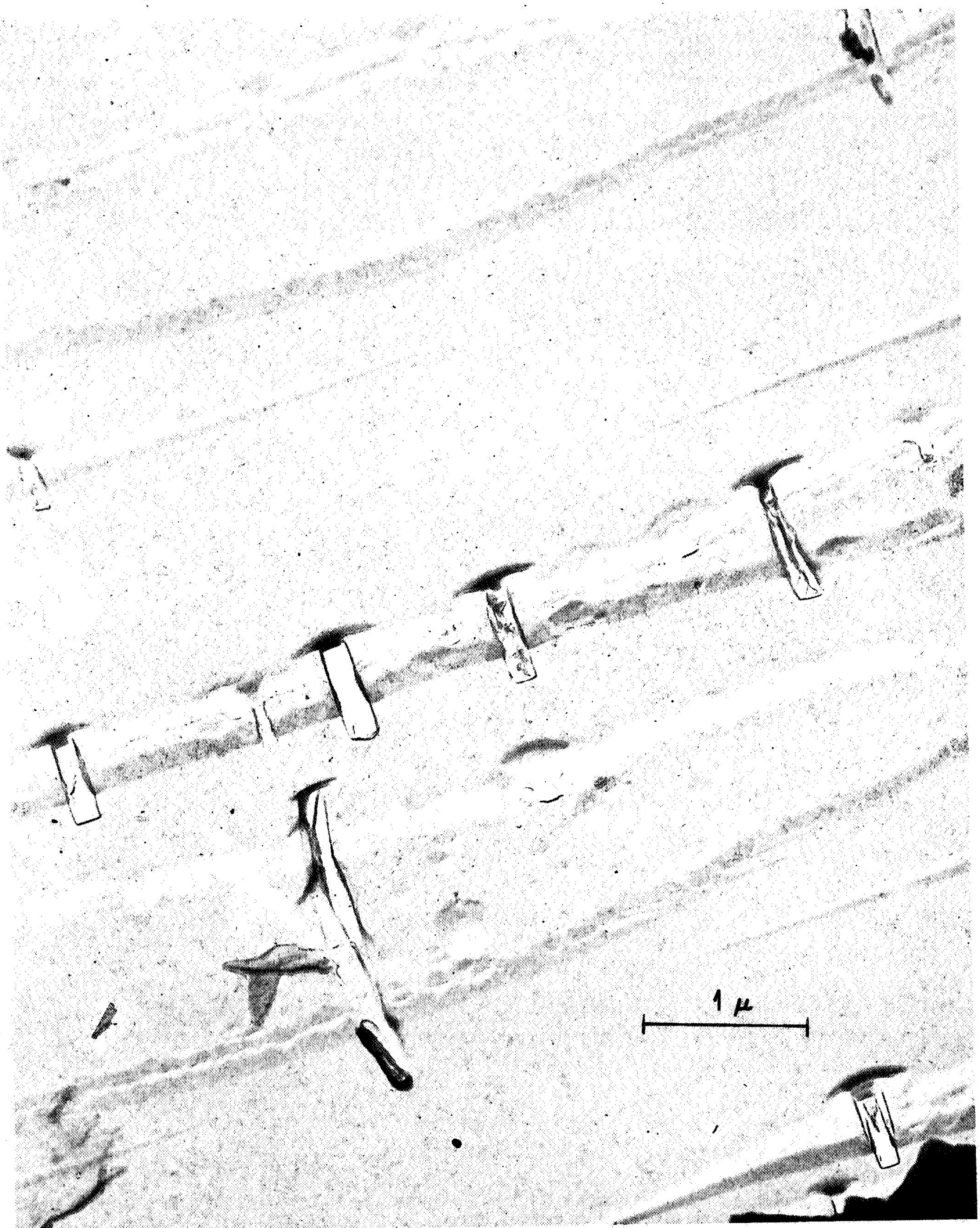

FIGURE 6

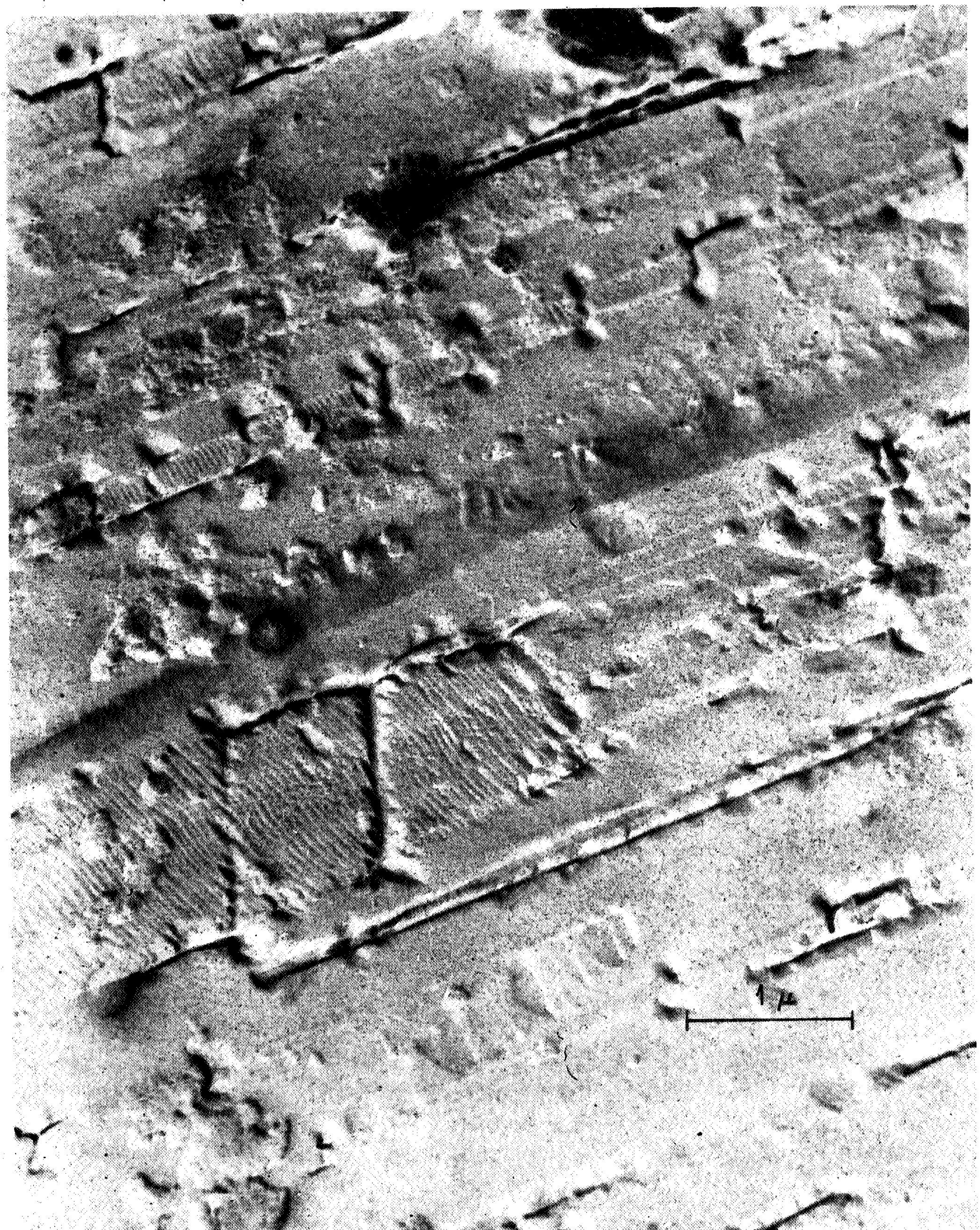

FIGURE 7

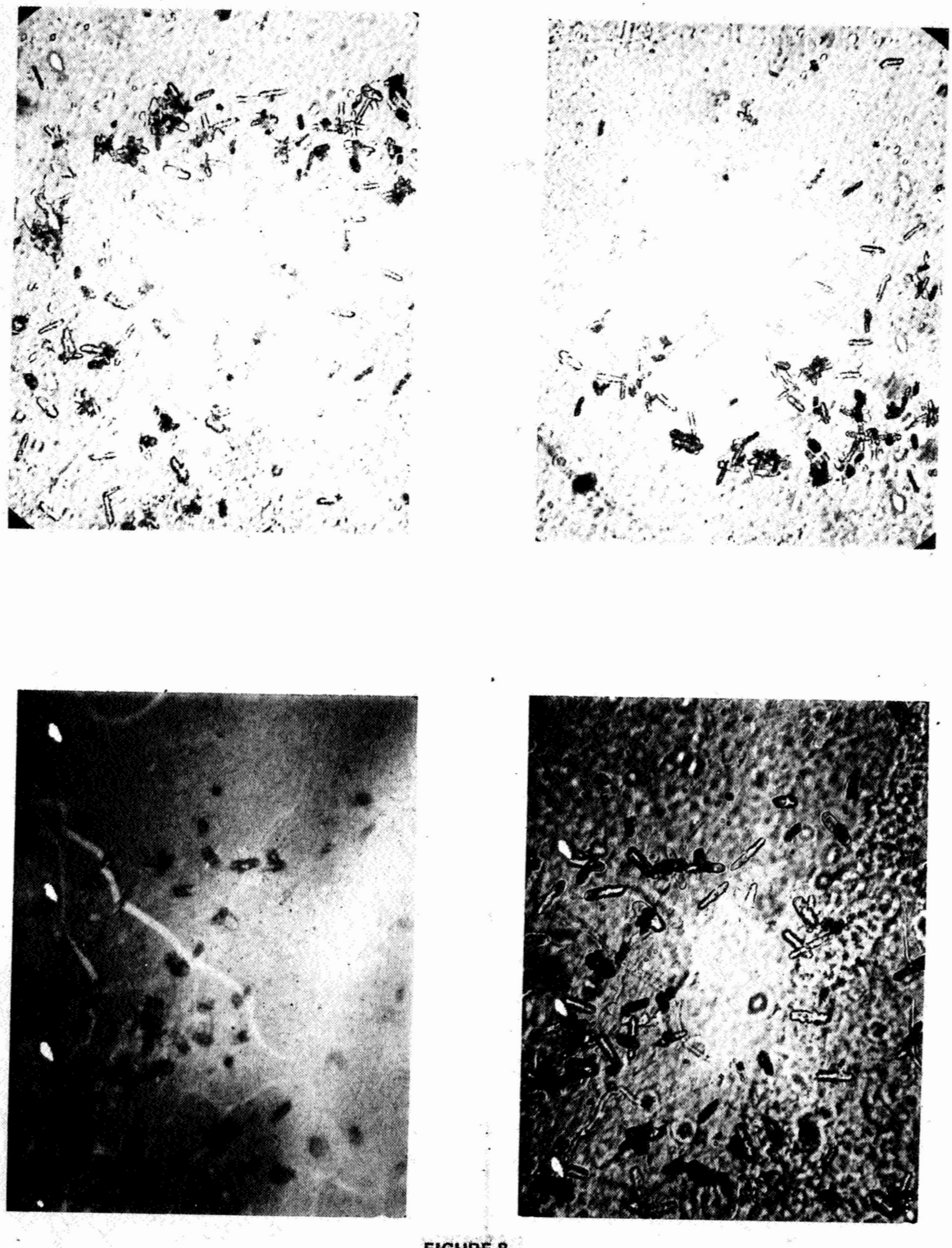

FIGURE 8